

Docket No.: 0365-0627PUS1  
(PATENT)

*IN THE UNITED STATES PATENT AND TRADEMARK OFFICE*

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In re Patent Application of:  
Hannu Mikkonen, *et al.*

Application No.: 10/532,398

Confirmation No.: 1581

Filing Date: October 12, 2005

Art Unit: 1623

For: NEW STARCH-BASED COMPOSITION AND  
PROCESS FOR THE PRODUCTION  
THEREOF

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Examiner: LAU, Jonathan S.

**DECLARATION UNDER 37 CFR 1.132**

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

1. I, Dr. Soili Hellevi Peltonen, declare as follows:

2. I graduated in 1969 with a Bachelors of Science from the University of Oulu, in Finland. I then obtained a Masters of Science from the University of Oulu in 1971, specifically studying organic chemistry as well as mathematics and physics. In 1976, I obtained my doctorate (Lic.Phil.) in organic chemistry from the University of Oulu concentrating my studies on polyphenols of spruce and pine bark.

GMM/LTP/la

3. In the 1970s and early 1980s I was employed variously as: a part-time teacher at the University of Oulu, studying polyphenolic compounds of wood bark (fractionation, characterisation and derivatization) as well as studying terpenes and oxetanes, a Research Associate at the University of Oulu; and a Research Associate at the Academy of Finland.

4. From 1981 through 1989, I was employed by Veitsiluoto Ltd., first as a Laboratory Manager and then as a Research Manager. My responsibilities involved Research and Development, Quality Control and Customer Service for Product Groups such as tall oil and turpentine distillates, adhesive tackifiers, binders for printing inks, polymerisation emulsifiers, rosin based paper sizes and SB-latices.

5. From 1989 until 1997, I was the Research and Development Manager at Primalco Ltd. I was primarily responsible for research and development of starch derivatives, starch based adhesives, tableting excipients, bioplastics, and water dispersions of bio-polymers, lactic acid polymers, and biomass fractionation.

6. From 1997 to the present, I have been the Team Leader, Customer Manager, and Research Coordinator for Valtion teknillinen tutkimuslaitos (VTT). From 1997 through 2006, I was responsible for research on biopolymers and natural polymers, and was also the Manager of Rajamäki pilot hall. Further, I was responsible for research and preparation of conductive polymers in co-operation with a customer. From 2007 to the present, I have been the Customer Manager in Chemistry and the Environment and Research Coordinator in Sustainable Chemistry.

7. I am familiar with the prosecution history of U.S. Patent Application No. 10/532,398, of which I am one of the named inventors.

8. I am aware of the Examiner's rejection of claims 1, 5-12, 14, 15, and 22-30 as allegedly unpatentable over U.S. Patent No. 3,346,558 to Roth ("Roth") in view of Leitheiser *et al.*, *Ind. Eng. Chem. Res. Dev.*, 1966, 5:276-282, ("Leitheiser"). In my considered opinion, these prior art rejections are not justified. Therefore, the following testing and discussion are provided.

9. The closest prior art is Roth, which discloses a process for preparing polyol-glycosides comprising reacting starch, polyol, and acid at an elevated pressure using a screw-type extruder. Comparative testing has been carried out with respect to the disclosure of Roth and a method according to the present application. More specifically, testing has been conducted to demonstrate how well the preferred acid of Roth, para-toluene sulfonic acid, works during reactive extrusion, as compared to a phosphorous-containing acid of the present invention, exemplified by hypophosphorous acid.

10. In a test procedure, 20 g of acetylated starch and ethylene glycol (15 w-% of starch acetate) and 0.5 g of catalyst were melt-mixed at 180 °C using a compounder (DACA Instruments). In two first experiments, a material was prepared using hypophosphorous acid as a catalyst, and two latter experiments, a material was prepared using para-toluene sulphonic acid as a catalyst. Two samples were prepared with both catalysts, the mixing times employed for the

samples being 5 min and 10 min, respectively. The materials obtained formed transparent, plastic-like products during compounding. The material with the para-toluene sulphonic acid as catalyst was somewhat darker in color. For analyzing the color stability in a melt state, samples of both materials were placed in an oven for 6 h. The oven temperature was 160 °C.

11. After heating for the indicated period of time, the color of the materials was visually examined and photographs were taken of the samples. Enclosed are copies of the photographs, *see* Exhibit A and Exhibit B. As is apparent from the Exhibits, the color of the material prepared using para-toluene sulphonic acid as a catalyst was very dark, but the color of the material prepared with a catalyst according to the present invention did not change during heating.

12. Further, the molecular weights of the samples were determined after preparation and after heat-treatment. The molecular masses of the materials are shown in the enclosed analysis data both after the transglycosylation reaction and after a 6 hour heating at 160 °C. *See* Exhibit C, and Exhibit E, which describes the material using the para-toluene before and after heat treatment, respectively. *See also* Exhibit D and Exhibit F, which describes the material using the phosphorus-containing acid catalyst encompassed by the claimed methods before and after heat treatment, respectively.

13. The molecular weight measurements show that para-toluene sulfonic acid results in strong degradation, *see* Exhibits C and D. The molecular masses of the materials are shown in the enclosed analysis data both after the transglycosylation reaction and after a 6 hour heating at 160 °C.

14. When using hypophosphorous acid, a bimodal molecular weight distribution is obtained, the value of the higher molecular weight range being weight average ( $M_w$ ) = 1,159,987 and numerical average ( $M_n$ ) = 668,684 and for the lower molecular weight range:  $M_w$  = 119,765 and  $M_n$  = 97,398, *see* Exhibit E.

15. Using para-toluenesulfonic acid, the values were  $M_w$  = 36,532 and  $M_n$  23,343, *see* Exhibit C. No bimodality is observed.

16. As is apparent from the Exhibits, para-toluene sulfonic acid results in a much lower molecular weight than that observed with hypophosphorous acid.

17. When the products were heated for 6 hours, the molecular weights were lowered for both different kinds of material, but the molecular weights of the materials encompassed by the claimed methods remained much higher. For the material obtained using hypophosphorous acid:  $M_w$  = 174,508 and  $M_n$  = 80,620, *see* Exhibit F. After heating, the material encompassed by the claimed methods was monomodal. For para-toluenesulfonic acid catalyzed samples, the molecular weights after heating were very low, viz.  $M_w$  = 8,426 and  $M_n$  = 5,315, *see* Exhibit D.

18. As far as use of the product as an adhesive, the present invention provides a product with a suitable molecular weight distribution in view of the fact that polymeric molecules are present having both a high molecular weight and a low molecular weight. The portion having high molecular weight gives properties of strength and film-forming and the lower weight portion reduces viscosity. No such distribution can be seen for the product obtained with para-toluene sulfonic acid; rather the material has inferior properties.

19. The present materials are suitable as hot melt adhesives; they have excellent properties also after extended heating as shown by the attached results. They maintain a high molecular weight and the color is unchanged. By contrast, the product according to the Roth reference cannot be used as adhesive. It becomes brittle after heating; viscosity drops. Brittleness will cause the glue joint to yield under stress. The material according to the present invention is instead viscous and flexible, as required for a good adhesive.

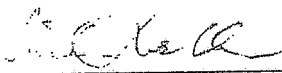
20. It is apparent that the present invention is distinct from the references cited by the Examiner, which relate to technologies that are significantly different from that of the present invention.

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STATEMENT UNDER 18 U.S.C. § 1001

I hereby declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001, of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

By: 

Date: Oct. 13, 2010

Dr. Soili Hellevi Peltonen